

|     |             |             |            |           |
|-----|-------------|-------------|------------|-----------|
| N1A | 0.1767 (1)  | -0.3136 (1) | 0.7473 (1) | 0.042 (1) |
| C2A | 0.1743 (1)  | -0.2358 (1) | 0.6650 (1) | 0.037 (1) |
| C3A | 0.0415 (1)  | -0.2274 (1) | 0.5512 (1) | 0.032 (1) |
| C4A | -0.0918 (1) | -0.3025 (1) | 0.5247 (1) | 0.051 (1) |
| C5A | -0.0896 (1) | -0.3808 (1) | 0.6103 (1) | 0.063 (1) |
| C6A | 0.0466 (1)  | -0.3836 (1) | 0.7208 (1) | 0.051 (1) |

Table 4. Geometric parameters ( $\text{\AA}$ ,  $^\circ$ ) for (II)

|           |           |             |           |
|-----------|-----------|-------------|-----------|
| C11—C6    | 1.725 (1) | O1—C7       | 1.222 (1) |
| C5—F1     | 1.342 (1) | N2—C7       | 1.349 (1) |
| N1—C2     | 1.335 (1) | N2—C3A      | 1.408 (1) |
| N1—C6     | 1.308 (1) | N1A—C2A     | 1.341 (1) |
| C2—C3     | 1.372 (1) | N1A—C6A     | 1.318 (1) |
| C3—C4     | 1.377 (1) | C2A—C3A     | 1.391 (1) |
| C3—C7     | 1.506 (1) | C3A—C4A     | 1.379 (1) |
| C4—C5     | 1.369 (1) | C4A—C5A     | 1.373 (1) |
| C6—C5     | 1.368 (1) | C5A—C6A     | 1.376 (1) |
| C2—N1—C6  | 117.2 (1) | O1—C7—C3    | 119.1 (1) |
| C3—C2—N1  | 124.4 (1) | C3—C7—N2    | 117.5 (1) |
| C2—C3—C4  | 117.4 (1) | O1—C7—N2    | 123.3 (1) |
| C2—C3—C7  | 125.4 (1) | C7—N2—C3A   | 126.6 (1) |
| C4—C3—C7  | 117.3 (1) | C2A—N1A—C6A | 119.0 (1) |
| C3—C4—C5  | 118.3 (1) | N1A—C2A—C3A | 122.5 (1) |
| F1—C5—C4  | 120.7 (1) | N2—C3A—C2A  | 124.7 (1) |
| F1—C5—C6  | 119.3 (1) | C4A—C3A—N2  | 117.8 (1) |
| C4—C5—C6  | 120.0 (1) | C2A—C3A—C4A | 117.5 (1) |
| N1—C6—C11 | 117.9 (1) | C3A—C4A—C5A | 119.6 (1) |
| C11—C6—C5 | 119.4 (1) | C4A—C5A—C6A | 119.3 (1) |
| C5—C6—N1  | 122.8 (1) | N1A—C6A—C5A | 122.1 (1) |

Dispersion corrections and absorption coefficients were taken from *International Tables for Crystallography*, 1992, Vol. C, Tables 6.1.1.4 and 4.2.6.8, and 4.2.4.2, respectively.

For both compounds, data collection: XSCANS (Siemens, 1991); cell refinement: XSCANS; data reduction: XSCANS; program(s) used to solve structures: SHELXS86 (Sheldrick, 1985). Program(s) used to refine structures: SHELXL93 (Sheldrick, 1993) for (I); SHELXS86 for (II). Molecular graphics: SHELXTL-Plus (Sheldrick, 1992) for (I); XP (Siemens, 1990) for (II). Software used to prepare material for publication: SHELXTL-Plus for (I); SHELXS86 for (II).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: CR1196). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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*Acta Cryst.* (1996). **C52**, 441–444

## A Chiral Mesogenic Compound

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(Received 10 May 1995; accepted 26 June 1995)

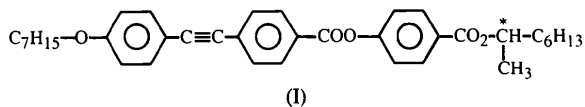
## Abstract

Single-crystal X-ray analysis has been carried out for 4-(1-methylheptyloxycarbonyl)phenyl 4-heptyloxytolane-4'-carboxylate [C<sub>7</sub>-tolane, C<sub>37</sub>H<sub>44</sub>O<sub>5</sub>, tolane = 1,1'-(1,2-ethylenediyl)bisbenzene]. The crystal has a smectic C-like layer structure composed of largely bent molecules; the chain of the chiral group is quasi-perpendicular (86°) to the core moiety. The molecular arrangement is similar to that of 4-[(S)-1-methylheptyloxycarbonyl]phenyl 4'-octyloxybiphenyl-4-carboxylate and agrees with the data of the *n* = 8 homologue (C8-tolane) antiferroelectric mesophase.

## Comment

In a number of crystal structures of mesogenic materials, good correlations have been established between the crystal organization and the mesomorphic smectic arrangement. In the case of ferroelectric materials it has been shown (Hori & Ohashi, 1991, 1993; Hori & Endo, 1993) that the molecular packing in the smectic-like solid layers is controlled by the arrangement of the polar groups. In a recent paper, Hori, Kawahara & Ito (1993) described the crystalline structure of several antiferroelectric materials. The most striking behaviour in the molecular structure is that the chain of the chiral groups is almost perpendicular to the core moiety; moreover,

these chains face each other between the neighbouring layers, the corresponding arrangement being antiferroelectric. This antiferroelectric ordering differs strongly from the usual model of such a phase in liquid crystals (*i.e.* antiparallel ordering of the ferroelectric dipoles in alternate layers) in that the alternation of dipoles is observed within each layer. The chemical formula of the studied compound, C7-tolane (I), is given below.



This compound presents a very rich polymorphism:  $K 92.3 \rightarrow S_{JA}^* (69.6) \rightarrow S_{IA}^* (73.7) \rightarrow S_{CA}^* (87.5) \rightarrow S_{CF11}^* (88.3) \rightarrow S_{CF12}^* (90) \rightarrow S_C^* 96.1 \rightarrow S_{C\alpha}^* \rightarrow 98.4 \rightarrow S_A 136 \rightarrow I$ , where the antiferroelectric phases ( $S_{CA}^*$ ,  $S_{JA}^*$  and  $S_{IA}^*$ ) and the ferroelectric phases ( $S_{CF11}$  and  $S_{CF12}$ ) are monotrope. The aim of this paper is to explore new chemical compounds in which antiferroelectric phases have been observed and to determine the crystalline ordering.

A drawing of the molecule and the atomic labelling are presented in Fig. 1. Note the rather high  $B_{eq}$  factors of the lateral alkyl chain (atoms C35–C42) illustrated in Fig. 1 and shown in Table 2. The three phenyl groups are designated as  $\emptyset 1$  (atoms C1–C6),  $\emptyset 2$  (C10–C15) and  $\emptyset 3$  (C20–C25). The  $\emptyset 1$ ,  $\emptyset 2$  and  $\emptyset 3$  phenyl groups are perfectly planar. The tolane central group is

roughly planar and makes an angle of  $60.8(1)^\circ$  with  $\emptyset 1$ . This planarity of the tolane group has been observed in other compounds (Cotrait, Destradé & Gasparoux, 1977). The  $\emptyset 1$ – $\emptyset 2$ ,  $\emptyset 1$ – $\emptyset 3$  and  $\emptyset 2$ – $\emptyset 3$  mean-plane angles are  $62.0(2)$ ,  $60.4(2)$  and  $4.7(2)^\circ$ , respectively; this is due to the C8–O7–C4–C3 torsion angle of  $-63.2^\circ$ . The heptyloxy chain is fully extended (atoms O18, C19, C26–C31) and perfectly planar; the hexyl lateral chain (C35–C42) is also *trans* and perfectly planar. All bond lengths and bond angles are as expected. Most torsion angles are close to either  $180$  or  $0^\circ$ ; the interesting ones are C3–C4–O7–C8  $-63.2(4)$ , C5–C4–O7–C8  $118.5(4)$ , C19–O18–C23–C22  $-175.3(4)$ , O33–C35–C37–C38  $-70.7(5)$ , C36–C35–C37–C38  $171.4(5)$ , C23–O18–C19–C26  $175.9(3)$ , C32–O33–C35–C36  $-92.9(4)$ , C32–O33–C35–C37  $147.6(4)^\circ$ .

The main intramolecular distances are as follows:  $d(C31 \cdots C35) = 28.864(8) \text{ \AA}$  for the full length of the molecule;  $d(C35 \cdots C42) = 7.35(1) \text{ \AA}$  for the alkyl branch;  $d(O18 \cdots C31) = 8.725(8) \text{ \AA}$  for the terminal heptyloxy chain;  $d(O18 \cdots C32) = 18.767(6) \text{ \AA}$  for the central core.

The projection of the structure along  $0y$  onto the  $xOz$  plane is shown in Fig. 2. The directions of the molecular central cores (O18–C32) are quasi-parallel with a mean distance close to  $5 \text{ \AA}$  forming sheets parallel to the  $yOz$  plane. The heptyloxy chains (O18–C31) make an angle close to  $30^\circ$  with the  $xOz$  plane and the terminal chiral chains (C35–C42) make an angle close to  $13^\circ$  with the  $xOz$  plane. The chiral side

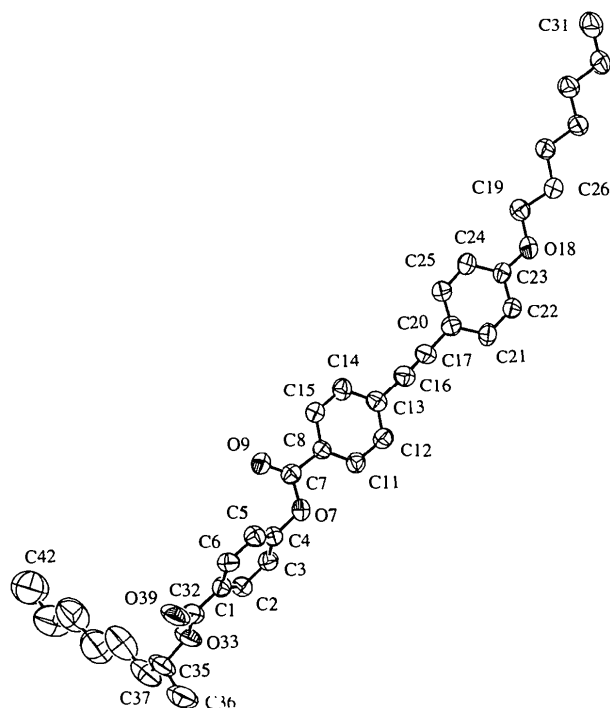


Fig. 1. Drawing of the molecule showing the atomic labelling. Displacement ellipsoids are drawn at the 50% probability level.

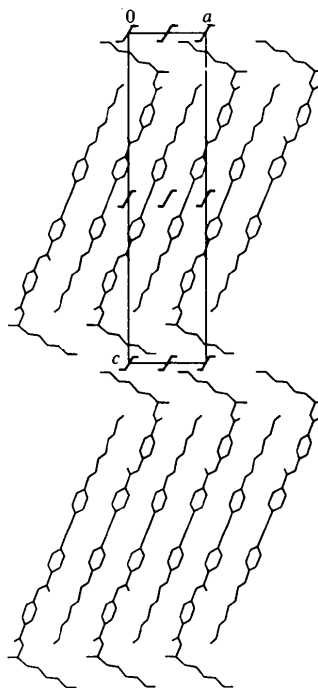


Fig. 2. Projection of the structure along the  $y$  axis onto the  $xOz$  plane.

chain (C35–C42) makes an angle of *ca* 86° with the direction of the core. This feature is mainly due to the torsion around the O33–C35 bond. As for the 4-[(*S*)-1-methylheptyloxycarbonyl]phenyl 4'-octyloxybiphenyl-4-carboxylate structure (Hori & Endo 1993), the present structure shows a smectic-like arrangement. The layer thickness is *ca* 36 Å and the tilt angle (angle between the central core and the normal to the sheet) is close to 23°. These values can be compared with those found for the C8-tolane (Cluzeau *et al.*, 1995) in the antiferroelectric phase (37 Å thickness and 19° for the tilt angle). The crystal cohesion is assumed to be almost uniquely due to weak van der Waals forces; this is in agreement with the relatively low density of 1.128 g cm<sup>-3</sup>. No particular interactions were noticed between adjacent layers. Nevertheless, there is a strong interaction between O34(*x*, *y*, *z*) and H336(*x*, 1 + *y*, *z*) (the H atom bonded to C36) with a distance of 2.24 (1) Å.

For the studied compound, the antiferroelectric chiral smectic phase does not follow the crystalline phase; however, the more-ordered intermediate phases are also antiferroelectric phases. We suppose that the molecular arrangement is slightly modified and then the crystal structure prefigures the antiferroelectric mesophase. The above remarks regarding the layer thickness and the tilt angle for C8-tolane reinforce our view.

## Experimental

Compound (I) was recrystallized from methanol/chloroform.

### Crystal data

C<sub>37</sub>H<sub>44</sub>O<sub>5</sub>  
*M<sub>r</sub>* = 568.76  
 Monoclinic  
*P*2<sub>1</sub>  
*a* = 8.467 (1) Å  
*b* = 5.526 (1) Å  
*c* = 35.792 (7) Å  
 $\beta$  = 90.29 (1)°  
*V* = 1674.5 Å<sup>3</sup>  
*Z* = 2  
*D<sub>x</sub>* = 1.128 Mg m<sup>-3</sup>

Cu K $\alpha$  radiation  
 $\lambda$  = 1.54178 Å  
 Cell parameters from 25 reflections  
 $\theta$  = 20–42°  
 $\mu$  = 0.591 mm<sup>-1</sup>  
*T* = 293 K  
 Prism  
 0.5 × 0.3 × 0.2 mm  
 Colourless

### Data collection

Enraf–Nonius CAD-4 diffractometer  
 $\omega/2\theta$  scans  
 Absorption correction:  $\psi$  scans (SDP; B. A. Frenz & Associates Inc., 1982)  
*T<sub>min</sub>* = 0.937, *T<sub>max</sub>* = 0.998  
 3460 measured reflections  
 2747 independent reflections  
 2229 observed reflections  
 [*I* > 2 $\sigma$ (*I*)]

*R<sub>int</sub>* = 0.96  
 $\theta_{\max}$  = 60°  
*h* = -9 → 9  
*k* = 0 → 6  
*l* = 0 → 40  
 3 standard reflections monitored every 120 reflections  
 frequency: 90 min  
 intensity decay: none

### Refinement

Refinement on *F*  
*R* = 0.064  
*wR* = 0.074  
*S* = 1.70  
 2229 reflections  
 555 parameters  
 All H-atom parameters refined  
 $w = 2.5/[\sigma^2(F) + 0.00136F^2]$

( $\Delta/\sigma$ )<sub>max</sub> = 0.15  
 $\Delta\rho_{\max} = 0.30 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\min} = -0.20 \text{ e \AA}^{-3}$   
 Extinction correction: none  
 Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.3.1)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å<sup>2</sup>)

|     | <i>x</i>    | <i>y</i>    | <i>z</i>    | <i>B<sub>eq</sub></i> |
|-----|-------------|-------------|-------------|-----------------------|
| C1  | 0.2960 (5)  | 0.2157 (9)  | 0.2156 (1)  | 4.6 (5)               |
| C2  | 0.3313 (5)  | 0.0322 (9)  | 0.2397 (1)  | 5.0 (5)               |
| C3  | 0.2742 (5)  | 0.0323 (9)  | 0.2761 (1)  | 5.0 (5)               |
| C4  | 0.1837 (4)  | 0.2204 (8)  | 0.2871 (1)  | 4.3 (5)               |
| C5  | 0.1468 (5)  | 0.4125 (8)  | 0.2642 (1)  | 4.5 (5)               |
| C6  | 0.2032 (5)  | 0.4112 (9)  | 0.2280 (1)  | 4.7 (5)               |
| O7  | 0.1245 (3)  | 0.2317 (6)  | 0.32417 (7) | 4.8 (3)               |
| C8  | 0.0245 (4)  | 0.0574 (8)  | 0.3349 (1)  | 4.2 (4)               |
| O9  | -0.0114 (3) | -0.1135 (7) | 0.31539 (8) | 5.5 (4)               |
| C10 | -0.0374 (4) | 0.0998 (8)  | 0.3734 (1)  | 4.0 (4)               |
| C11 | 0.0085 (5)  | 0.2944 (8)  | 0.3950 (1)  | 4.5 (5)               |
| C12 | -0.0503 (5) | 0.3182 (8)  | 0.4310 (1)  | 4.7 (5)               |
| C13 | -0.1543 (4) | 0.1489 (9)  | 0.4452 (1)  | 4.5 (5)               |
| C14 | -0.2038 (5) | -0.0432 (9) | 0.4227 (1)  | 5.2 (5)               |
| C15 | -0.1449 (5) | -0.0681 (9) | 0.3875 (1)  | 4.7 (5)               |
| C16 | -0.2183 (4) | 0.1652 (9)  | 0.4827 (1)  | 5.0 (5)               |
| C17 | -0.2716 (4) | 0.1670 (9)  | 0.5128 (1)  | 4.7 (5)               |
| O18 | -0.5018 (3) | 0.1536 (6)  | 0.65893 (7) | 5.1 (3)               |
| C19 | -0.5889 (5) | -0.0472 (9) | 0.6728 (1)  | 4.8 (5)               |
| C20 | -0.3361 (4) | 0.1601 (8)  | 0.5500 (1)  | 4.4 (5)               |
| C21 | -0.2973 (5) | 0.3428 (9)  | 0.5761 (1)  | 5.0 (5)               |
| C22 | -0.3564 (5) | 0.3369 (9)  | 0.6117 (1)  | 4.8 (5)               |
| C23 | -0.4503 (4) | 0.1465 (8)  | 0.6229 (1)  | 4.1 (4)               |
| C24 | -0.4903 (5) | -0.0338 (8) | 0.5977 (1)  | 4.9 (5)               |
| C25 | -0.4330 (5) | -0.0237 (9) | 0.5614 (1)  | 4.9 (5)               |
| C26 | -0.6224 (5) | -0.0073 (9) | 0.7135 (1)  | 4.8 (5)               |
| C27 | -0.7119 (5) | -0.2214 (8) | 0.7293 (1)  | 4.9 (5)               |
| C28 | -0.7480 (5) | -0.2009 (9) | 0.7708 (1)  | 5.2 (5)               |
| C29 | -0.8236 (5) | -0.4339 (9) | 0.7860 (1)  | 5.6 (5)               |
| C30 | -0.8637 (6) | -0.419 (1)  | 0.8271 (1)  | 6.4 (5)               |
| C31 | -0.9415 (6) | -0.639 (1)  | 0.8418 (1)  | 7.7 (5)               |
| C32 | 0.3575 (5)  | 0.2030 (9)  | 0.1767 (1)  | 5.5 (5)               |
| O33 | 0.3168 (4)  | 0.3892 (7)  | 0.15568 (8) | 7.0 (5)               |
| O34 | 0.4366 (5)  | 0.0428 (8)  | 0.16574 (9) | 8.6 (5)               |
| C35 | 0.3844 (6)  | 0.401 (1)   | 0.1180 (1)  | 7.9 (5)               |
| C36 | 0.5328 (7)  | 0.550 (1)   | 0.1198 (2)  | 9.9 (6)               |
| C37 | 0.2638 (7)  | 0.519 (1)   | 0.0933 (2)  | 10.3 (7)              |
| C38 | 0.1249 (8)  | 0.349 (1)   | 0.0870 (2)  | 12.5 (5)              |
| C39 | 0.0121 (9)  | 0.391 (1)   | 0.0591 (2)  | 14.3 (5)              |
| C40 | -0.1201 (9) | 0.205 (1)   | 0.0545 (2)  | 13.0 (4)              |
| C41 | -0.233 (1)  | 0.284 (1)   | 0.0284 (3)  | 15.8 (8)              |
| C42 | -0.3677 (9) | 0.099 (1)   | 0.0257 (3)  | 15.1 (9)              |

Table 2. Selected geometric parameters (Å, °)

|         |           |         |           |
|---------|-----------|---------|-----------|
| C1–C2   | 1.363 (6) | C19–C26 | 1.502 (6) |
| C1–C6   | 1.409 (6) | C20–C21 | 1.413 (6) |
| C1–C32  | 1.491 (6) | C20–C25 | 1.369 (6) |
| C2–C3   | 1.392 (6) | C21–C22 | 1.372 (6) |
| C3–C4   | 1.351 (6) | C22–C23 | 1.380 (6) |
| C4–C5   | 1.376 (6) | C23–C24 | 1.385 (6) |
| C4–O7   | 1.422 (5) | C24–C25 | 1.390 (6) |
| C5–C6   | 1.383 (6) | C26–C27 | 1.516 (6) |
| O7–C8   | 1.340 (5) | C27–C28 | 1.522 (6) |
| C8–O9   | 1.212 (5) | C28–C29 | 1.539 (6) |
| C8–C10  | 1.495 (6) | C29–C30 | 1.514 (7) |
| C10–C11 | 1.379 (6) | C30–C31 | 1.481 (7) |
| C10–C15 | 1.396 (6) | C32–O33 | 1.319 (6) |

|                 |            |                 |           |  |
|-----------------|------------|-----------------|-----------|--|
| C11—C12         | 1.390 (6)  | C32—O34         | 1.178 (6) | Cluzeau, P., Nguyen, H. T., Destrade, C., Isaert, N., Barois, P. & Babeau, A. (1995). <i>Mol. Cryst. Liq. Cryst.</i> In the press.         |
| C12—C13         | 1.383 (6)  | O33—C35         | 1.469 (6) | Cotrait, M., Destrade, C. & Gasparoux, H. (1977). <i>Mol. Cryst. Liq. Cryst.</i> <b>39</b> , 159–172.                                      |
| C13—C14         | 1.396 (6)  | C35—C36         | 1.503 (8) | Davies, K. (1983). <i>SNOOPI. Program for Drawing Crystal and Molecular Diagrams</i> . Chemical Laboratory, University of Oxford, England. |
| C13—C16         | 1.453 (6)  | C35—C37         | 1.497 (8) | Enraf–Nonius (1977). <i>CAD-4 Operations Manual</i> . Enraf–Nonius, Delft, The Netherlands.  |
| C14—C15         | 1.364 (6)  | C37—C38         | 1.521 (9) | Gilmore, C. J. (1984). <i>J. Appl. Cryst.</i> <b>17</b> , 42–46.   |
| C16—C17         | 1.170 (6)  | C38—C39         | 1.40 (1)  | Hori, K. & Endo, K. (1993). <i>Bull. Chem. Soc. Jpn.</i> <b>66</b> , 46–50.  |
| C17—C20         | 1.442 (6)  | C39—C40         | 1.53 (1)  | Hori, K., Kawahara, S. & Ito, K. (1993). <i>Ferroelectrics</i> , <b>147</b> , 91–94.   |
| O18—C19         | 1.423 (5)  | C40—C41         | 1.40 (1)  | Hori, K. & Ohashi, Y. (1991). <i>J. Mater. Chem.</i> <b>1</b> , 667–672.   |
| O18—C23         | 1.364 (5)  | C41—C42         | 1.53 (1)  | Hori, K. & Ohashi, Y. (1993). <i>Mol. Cryst. Liq. Cryst.</i> <b>203</b> , 171–176.   |
| C2—C1—C6        | 119.5 (4)  | C17—C20—C21     | 120.2 (4) | Sheldrick, G. M. (1976). <i>SHELX76. Program for the Solution of Crystal Structures</i> . University of Cambridge, England.                |
| C2—C1—C32       | 118.6 (4)  | C17—C20—C25     | 121.7 (4) |  |
| C6—C1—C32       | 121.9 (4)  | C21—C20—C25     | 118.1 (4) |  |
| C1—C2—C3        | 121.1 (4)  | C20—C21—C22     | 120.8 (4) |  |
| C2—C3—C4        | 118.2 (4)  | C21—C22—C23     | 120.1 (4) |  |
| C3—C4—C5        | 123.2 (4)  | O18—C23—C22     | 116.1 (4) |  |
| C3—C4—O7        | 120.6 (4)  | O18—C23—C24     | 123.9 (4) |  |
| C5—C4—O7        | 116.2 (4)  | C22—C23—C24     | 119.9 (4) |  |
| C4—C5—C6        | 118.4 (4)  | C23—C24—C25     | 119.6 (4) |  |
| C1—C6—C5        | 119.7 (4)  | C20—C25—C24     | 121.4 (4) |  |
| C4—O7—C8        | 117.5 (3)  | C19—C26—C27     | 110.2 (3) |  |
| O7—C8—O9        | 123.5 (4)  | C26—C27—C28     | 114.2 (3) |  |
| O7—C8—C10       | 112.1 (3)  | C27—C28—C29     | 111.6 (3) |  |
| O9—C8—C10       | 124.4 (4)  | C28—C29—C30     | 113.2 (4) |  |
| C8—C10—C11      | 122.7 (4)  | C29—C30—C31     | 113.8 (4) |  |
| C8—C10—C15      | 117.5 (3)  | C1—C32—O34      | 123.2 (4) |  |
| C11—C10—C15     | 119.9 (4)  | C1—C32—O33      | 113.9 (4) |  |
| C10—C11—C12     | 119.5 (4)  | C32—O33—C35     | 117.1 (4) |  |
| C11—C12—C13     | 120.5 (4)  | O33—C35—C36     | 108.3 (4) |  |
| C12—C13—C14     | 119.5 (4)  | O33—C35—C37     | 107.1 (4) |  |
| C12—C13—C16     | 122.6 (4)  | O34—C32—O33     | 122.9 (4) |  |
| C14—C13—C16     | 117.9 (4)  | C36—C35—C37     | 110.7 (5) |  |
| C13—C14—C15     | 120.0 (4)  | C35—C37—C38     | 110.1 (5) |  |
| C10—C15—C14     | 120.6 (4)  | C37—C38—C39     | 121.9 (6) |  |
| C13—C16—C17     | 176.8 (5)  | C38—C39—C40     | 117.6 (7) |  |
| C16—C17—C20     | 178.0 (5)  | C39—C40—C41     | 111.0 (7) |  |
| C19—O18—C23     | 118.4 (3)  | C40—C41—C42     | 109.8 (7) |  |
| O18—C19—C26     | 108.9 (3)  |                 |           |  |
| C3—C4—O7—C8     | −63.2 (4)  | C32—O33—C35—C36 | −92.9 (4) |  |
| C5—C4—O7—C8     | 118.5 (4)  | C32—O33—C35—C37 | 147.6 (4) |  |
| C23—O18—C19—C26 | 175.9 (3)  | O33—C35—C37—C38 | −70.7 (5) |  |
| C19—O18—C23—C22 | −175.3 (4) | C36—C35—C37—C38 | 171.4 (5) |  |

The structure was solved by direct methods, which led to the positions of almost all non-H atoms; the remaining atoms appeared after successive Fourier syntheses. The atomic parameters of non-H atoms were refined with full-matrix least squares. H atoms were located in their theoretical positions (refined isotropically) and followed the C atoms to which they are attached.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1977). Cell refinement: *CAD-4 Software*. Data reduction: *SDP* (B. A. Frenz & Associates Inc., 1982). Program(s) used to solve structure: *MITHRIL* (Gilmore, 1984). Program(s) used to refine structure: *SHELX76* (Sheldrick, 1976). Molecular graphics: *SNOOPI* (Davies, 1983). Software used to prepare material for publication: local programs.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, and complete geometry have been deposited with the IUCr (Reference: PA1193). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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*Acta Cryst.* (1996). **C52**, 444–447

## A 5,18:9,14-Di-*o*-benzeno-6,17:7,16:8,15-tri-methanobenzo[1'',2'':3,4;4'',5'':3',4']dicyclobuta[1,2-*b*:1',2'-*b'*]dianthracene Derivative

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(Received 6 January 1993; accepted 17 July 1995)

## Abstract

The phenylthiomethyl and trichloroacetyl groups of the title compound, 2-phenylthiomethyl-2-propenoic acid (5a $\alpha$ ,6 $\beta$ ,6a $\alpha$ ,6b $\beta$ ,7 $\alpha$ ,7a $\beta$ ,7b $\alpha$ ,8 $\beta$ ,8a $\alpha$ ,14a $\alpha$ ,15 $\beta$ ,15a $\alpha$ ,15b $\beta$ ,16 $\alpha$ ,16a $\beta$ ,16b $\alpha$ ,17 $\beta$ ,17a $\alpha$ )[6,6a,6b,7,7a,7b,8,8a,9,14,14a,15,15a,15b,16,16a,16b,17,17a,18-icosahydro-9-(trichloroacetoxymethyl)-5,18:9,14-di-*o*-benzeno-6,17:7,16:8,15-trimethanobenzo[1'',2'':3,4;4'',5'':3',4']dicyclobuta[1,2-*b*:1',2'-*b'*]dianthracen-5(5aH)-yl]methyl ester hemihydrate, C<sub>63</sub>H<sub>55</sub>Cl<sub>3</sub>O<sub>4</sub>S.0.5H<sub>2</sub>O, adopt a *cis* orientation with respect to each other about the hydrocarbon skeleton of the template. The average bond distances in the hydrocarbon skeleton are: Csp<sup>3</sup>—Csp<sup>3</sup> 1.54 (1), Csp<sup>3</sup>—Csp<sup>2</sup> 1.52 (1) and C—C aromatic 1.39 (1) Å.

## Comment

The main objective of the crystal analysis described in this paper was to ascertain the molecular structure and conformation of the title compound, (1). The crystal structure is composed of independent molecules of (1) separated by normal van der Waals distances. The asymmetric unit also contains a half molecule of water, lying close to an inversion centre and disordered.